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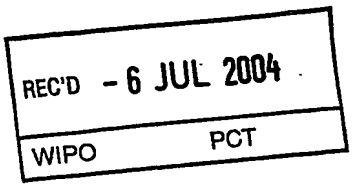

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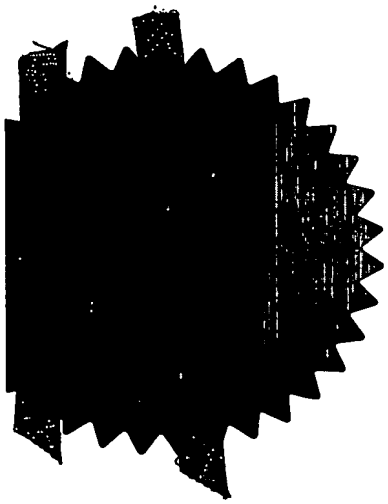

Signed Andrew Gerry

Dated 15 June 2004

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Patents Form 1/77

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The **Patent Office**

1/77

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet, from the Patent Office to help you fill in this form)

THE PATENT OFFICE
J
20 JUN 2003
NEWPORT

The Patent Office

Cardiff Road
Newport
Gwent NP9 1RH

20 JUN 2003

1. Your reference — CDK2110

2. Patent application number *(The Patent Office will fill in this part)* — 0314363.3

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

RHODIA CONSUMER SPECIALTIES LIMITED
Oak House, Reeds Crescent, Watford,
Hertfordshire, WD24 4QP,
and
THAMES WATER UTILITIES LIMITED
P.O. Box 234, Swindon, SN38 3TW

78 7032200

Patents ADP number *(if you know it)*

4412862006

20JUN03 E816504-5 D02806
P01/7700 0.00-0314363.3

If the applicant is a corporate body, give the country/state of its incorporation

England

4. Title of the invention — TREATMENT OF SEWAGE SLUDGE

5. Name of your agent *(if you have one)* — Barker Brettell

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

138 Hagley Road
Edgbaston
Birmingham
B16 9PW

Patents ADP number *(if you know it)* — 7442494002

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of Filing *(day/month/year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day/month/year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request *(Answer 'Yes' if:*
a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d)) — YES

Patents Form 1/77

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Continuation sheets of this form	-
Description	7 + 7
Claim(s)	2 + 2
Abstract	-
Drawing(s)	3 + 3

10. If you are also filing any of the following, state how many against each item.

Priority documents	-
Translations of priority documents	-
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	-
Request for preliminary examination *(Patents Form 9/77)*	1
Request for substantive examination *(Patents Form 10/77)*	-
Any other documents *(please specify)*	-

11. I/We request the grant of a patent on the basis of this application.

Signature Barker Brettell Date

Barker Brettell 19 June 2003

12. Name and daytime telephone number of person to contact in the United Kingdom

Colin D. Kinton Tel: 0121 456 1364

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TREATMENT OF SEWAGE SLUDGE

This invention relates to a method of treating sewage sludge and to a sludge treated by the aforesaid method.

The treatment of raw sewage generally includes a filtration stage (in which large solids and grit are removed) followed by a stage in which the aqueous phase is subjected to aerobic bacterial action to remove biodegradable substances. This latter stage involves "activated sludge" which is essentially a concentrated bacterial mass. Biodegradable substances need to be removed prior to the discharge of the aqueous phase into watercourses, e.g. rivers, as otherwise, the bacterial degradation of such substances in the river would consume dissolved oxygen resulting in fish deaths, odours and general degradation of the environment. During the degradation of the biodegradable substances, growth and multiplication of the bacteria occur, resulting in the accumulation of bacterial sludge requiring disposal.

Optionally, the excess sludge may be "digested" under anaerobic conditions where, essentially, the bacteria re-equilibrate under the new conditions to produce methane and reduce the biomass but, ultimately, there remains an irreducible mass of excess sludge which needs disposal. There are a number of methods of disposal, such as landfill and disposal at sea, both of which are disfavoured for environmental reasons. Alternatively, the excess sludge may be incinerated (expensive) or spread on to agricultural land and, in the latter case, the sludge can be used as a fertiliser/soil conditioner, which is a benefit.

Unfortunately, such sludge can contain significant concentrations of pathogens and, if so, the sludge requires disinfection to reduce to an environmentally-acceptable level any pathogenic organisms present,

before the disinfected sludge is spread to land. An indicator organism, used to quantify the pathogenic risk, is E.coli. For compliance with statutory provisions, the level of E.coli in the sludge must be reduced by 99% (i.e. a logarithmic reduction of 2) and the maximum acceptable level of E.coli in the treated sewage sludge is 10^5 per gram of dry sludge.

Bacterial reduction may be accomplished in a variety of ways including lime treatment (messy, requires significant capital investment and poses severe handling problems) heat treatment (very expensive) or merely leaving the sludge in storage till the bacterial level falls within the required limit. For the latter situation, the very large volumes of sludge involved at most sewage treatment works cannot usually be stored for the requisite time due to insufficient storage capacity. Installing sufficient capacity is either impractical due to space considerations or involves large capital expenditure.

In theory, an alternative method of reducing the bacterial content of the sludge would be to apply a disinfectant. However, disinfectants evaluated hitherto have been found to take relatively long periods to reduce the bacterial content to an acceptable level, thus creating storage demands beyond the resources of most sewage-treatment works.

We have found that the use of a phosphorus-containing compound (especially a phosphonium salt) on sewage sludge can bring about a reduction in the bacterial content of the sludge equivalent to a logarithmic decrease of at least 2 in a period of 24 hours.

Thus, the present invention provides a method of treating sewage sludge to reduce the bacterial content of said sludge, the method comprising the steps of:

(a) adding to the sludge an effective amount of a phosphorus-containing compound; and

(b) keeping the phosphorus-containing compound in contact with the sludge for sufficient time to reduce the bacterial content by an amount equivalent to a logarithmic reduction of at least 2 in a period of 24 hours.

Preferably, the sludge has undergone anaerobic digestion prior to step (a).

Preferably, the phosphorus-containing compound is a phosphonium compound, especially a tetrakis(hydroxyorgano) phosphonium salt.

Most preferably, the phosphonium compound is tetrakis(hydroxymethyl) phosphonium sulphate.

Alternatively, the phosphonium compound may be, for example, a tetrakis(hydroxymethyl) phosphonium halide, phosphate or acetate.

Again alternatively, the phosphorus-containing compound may be an alkyl-substituted phosphine, e.g. tris(hydroxymethyl) phosphine.

The amount of phosphorus-containing compound to be added to the sludge in step (a) of the method of the present invention is suitably up to 10000mg/l, preferably 250-1000mg/l, and especially 500-1000mg/l.

Alternatively, the amount of phosphorus-containing compound to be added to the sludge may be expressed relative to dry solids weight. Suitably, the amount to be added is up to about 20% by weight of dry solids.

Preferably, step (b) of the method of the present invention is carried out over a period of from 6 to 24 hours. The rate of addition of the phosphorus-containing compound and the rate of mixing are important in maximising the efficacy of the process. To maximise efficacy, both should be as short as practically possible.

The present invention further provides a sewage sludge which has been treated according to the method described hereinabove.

The present invention will be illustrated by way of the following Examples:

In the Examples, the phosphorus-containing compound used to treat sewage sludge was 75% w/w tetrakis(hydroxymethyl) phosphonium sulphate, available from Rhodia Consumer Specialties Limited. For the purposes of this patent specification, the product will be subsequently referred to as "Phosphonium Salt".

As a comparison, sewage sludges were treated with a second phosphorus-containing compound, dibromo-nitrilo-propionamide (DBNPA).

In each Example, the bacterium being observed was E.coli.

1.1 METHODOLOGY

The methodology adopted to evaluate biocide performance was by Quantitative Suspension Test (QST) using sterile anaerobic digester sludge as the QST medium, back-inoculated with E.coli cultures previously isolated from the sludge. In this way, a consistent chemical environment (sterile sludge) could be used in conjunction with a defined

bacterial challenge. This enables the provision of consistency between tests.

1.2 MICROBIOLOGICAL EVALUATIONS

Sterile sludge was prepared from raw sludge samples by autoclaving at 121°C for 20 minutes. The E.coli strains used in QST had been isolated from raw sludge samples.

QST were performed as follows:

- Sterile sludge (19ml) was dispensed into sterile, screw-cap, plastics universal bottles of nominal 30ml capacity.

- To each sample was added 0.5ml of a washed cell suspension of E.coli prepared from a 16-hour culture incubated at 44°C in Tryptose Soy Broth which had been centrifuged (14500 rpm for 10 min.) and re-suspended in sterile phosphate buffer (0.2M at pH 7.2). An inoculum of 0.5ml was sufficient to provide a final cell concentration of about 10^8 per ml in 20 ml of QST medium.

- Fresh stock solutions of the phosphorus-containing compounds were prepared in sterile phosphate buffer (0.2M at pH 7.2) at concentrations such that when 0.5ml was added to the QST medium (final volume 20ml) the desired final concentration of biocide was achieved.

- The QST medium was mixed thoroughly and held at 22°C for the duration of the test.

- At intervals during the test, samples (1.0ml) were removed from the QST medium and inoculated into the first tube of a dilution series containing MacConkey broth supplemented with sodium thiosulphate (5.0 g/l), to inactivate any residual biocide carried into the dilution series. This was carried out in duplicate.

- The remainder of the serial dilution (10 fold steps) was carried out in MacConkey Broth alone and tubes incubated at 44°C for 16 hours. The end point was scored as the highest dilution in the series to show a change in colour from purple to yellow and to have developed turbidity.

MacConkey Broth was selected as this medium contains the pH indicator Bromocresol Purple which changes from purple to yellow as the medium becomes acidic. This is a useful indirect indicator of microbial growth (organic acid production) where this cannot be scored by the development of turbidity in an initially clear medium. Because the sludge contains suspended solids the first 2 tubes of the dilution series instantaneously develop turbidity on the addition of the sludge. This precludes using turbidity alone as an indicator of microbial growth.

The biocides used in the evaluations are shown in the Table below.

BIOCIDE TYPE	ACTIVE INGREDIENT (ai)	PERCENT ai
Phosphonium Salt	THPS	75
DBNPA	DBNPA	98

EXAMPLES 1 to 3

The performance of Phosphonium Salt in the concentration range 250 to 100mg/l is illustrated in Figure 1 of the accompanying drawings.

Concentrations of 250 and 500 mg/l gave similar results with a fairly flat time/kill response over the first 6 hours contact time, followed by a progressive reduction in numbers to a total kill within 48 hours.

By contrast, the time/kill response at 1000mg/l was much improved. The time/kill response over the first 6 hours contact time was more progressive and total kill was achieved within 24 hours.

In the QST methodology used, the numbers of E.coli back-inoculated into sterile sludge slowly decreased spontaneously over time, as shown in Figure 2 of the accompanying drawings.

EXAMPLE 4

The performance of Phosphonium Salt compared to that of DBNPA, is shown in Figure 3 of the accompanying drawings. Both biocides were tested at an equal active-ingredient concentration of 500mg/l. DBNPA shows surprisingly poor anti-microbial performance, achieving only a 2.5 log reduction in numbers after 48 hours.

The foregoing Examples demonstrate the following characteristics of the present invention:

(a) Increasing the Phosphonium Salt concentration used in treatment from 500 to 1000mg/l gives a significant improvement in performance.

(b) In all of the treatments evaluated total kill was achieved.

(c) When compared with the performance of DBNPA, the performance of Phosphonium Salt was superior.

CLAIMS

1. A method of treating sewage sludge to reduce the bacterial content of said sludge, the method comprising the steps of:

(a) adding to the sludge an effective amount of a phosphorus-containing compound; and

(b) keeping the phosphorus-containing compound in contact with the sludge for sufficient time to reduce the bacterial content by an amount equivalent to a logarithmic reduction of at least 2 in a period of 24 hours.

2. A method according to Claim 1, in which the sludge has undergone anaerobic digestion prior to step (a).

3. A method according to Claim 1 or 2, in which the phosphorus-containing compound is an alkyl-substituted phosphine.

4. A method according to Claim 3, in which the phosphorus-containing compound is tris(hydroxymethyl) phosphine.

5. A method according to Claim 1 or 2, in which the phosphorus-containing compound is a phosphonium compound.

6. A method according to Claim 5, in which the phosphonium compound is a tetrakis(hydroxyorgano) phosphonium salt.

7. A method according to Claim 5 or 6, in which the phosphonium compound is tetrakis(hydroxymethyl) phosphonium sulphate.

8. A method according to Claim 5 or 6, in which the phosphonium compound is a tetrakis(hydroxymethyl) phosphonium halide, phosphate or acetate.

9. A method according to any one of the preceding claims, in which up to 10000mg of the phosphorus-containing compound per litre of sludge is added in step (a).

10. A method according to Claim 9, in which the amount of phosphorus-containing compound added to the sludge is in the range 250-1000mg/l.

11. A method according to Claim 9 or 10, in which the amount of phosphorus-containing compound added to the sludge is in the range 500-1000mg/l.

12. A method according to any one of Claims 1 to 8, in which up to about 20% by weight, relative to the dry solids content of the sludge, is added in step (a).

13. A method according to any one of the preceding claims, in which step (b) is carried out over a period of from 6 to 24 hours.

14. A method of treating sewage sludge, substantially as hereinbefore described with reference to the Examples.

15. A sewage sludge which has been treated according to the method of any one of the preceding claims.

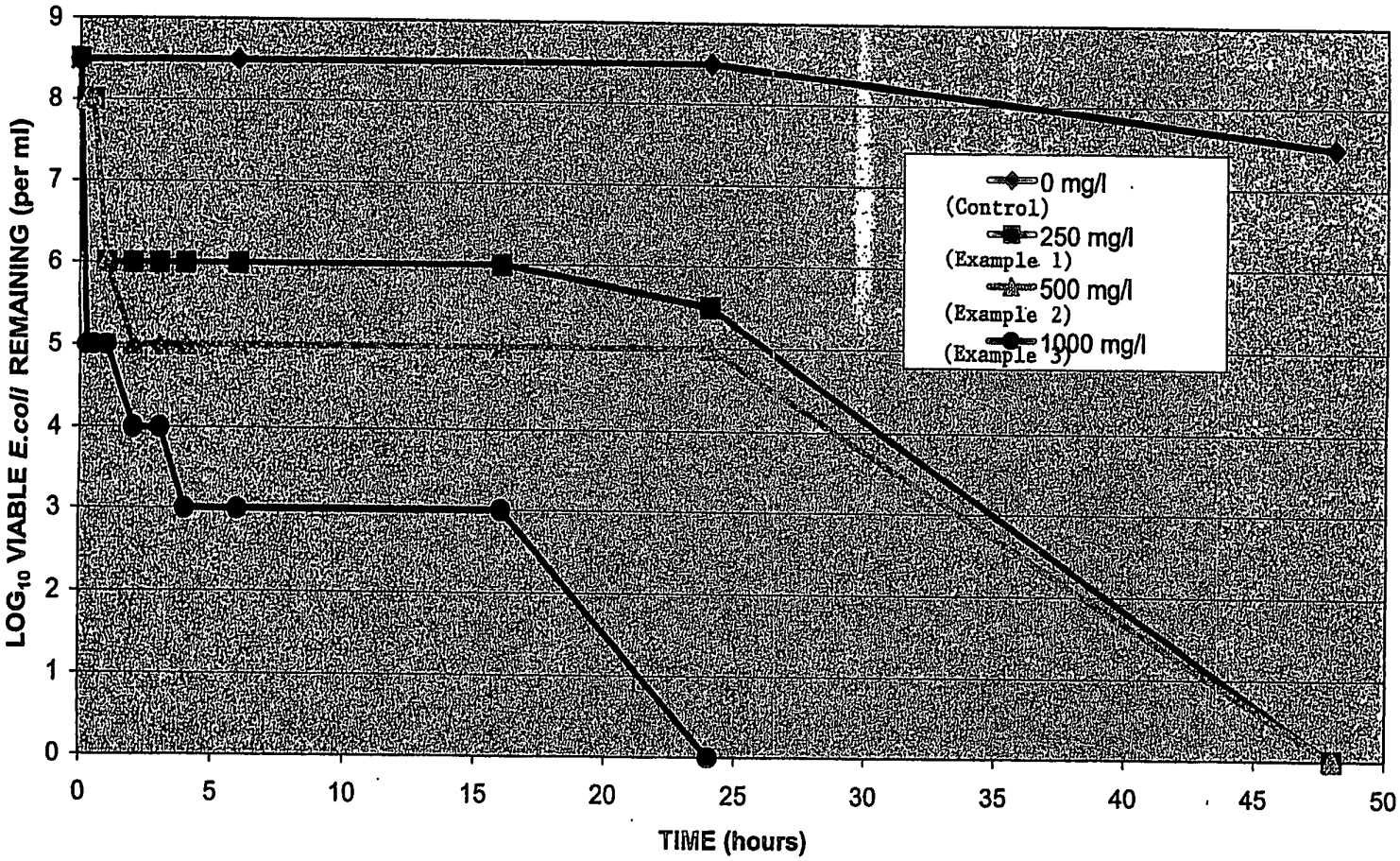
9
8
7
6
5
4
3
2
1
0
LOG10 VIABLE E.coli REMAINING (per ml)
0
5
10
15
20
25
30
35
40
45
50
TIME (hours)
0 mg/l
(Control)
250 mg/l
(Example 1)
500 mg/l
(Example 2)
1000 mg/l
(Example 3)

Fig. 1

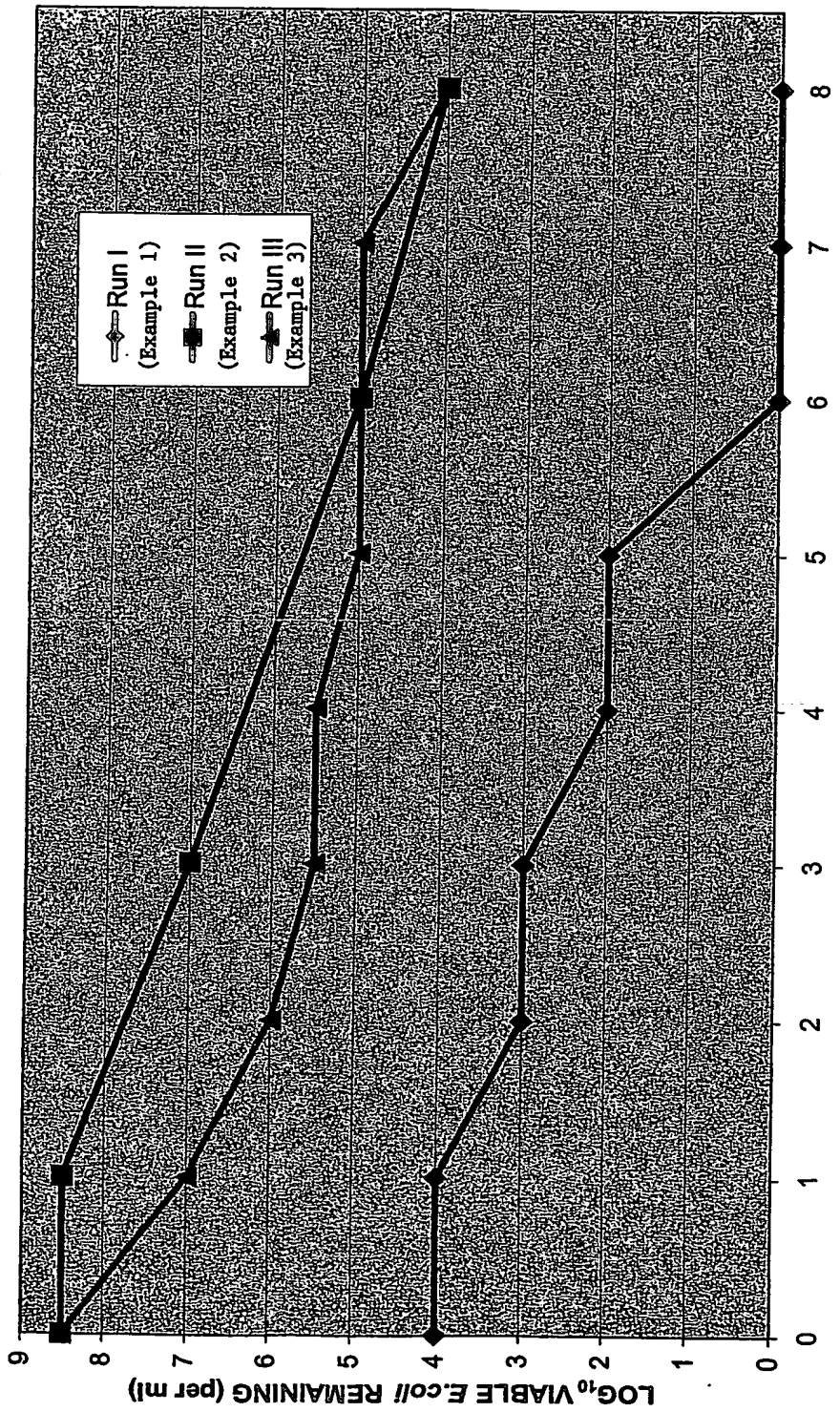
Run I
(Example 1)
Run II
(Example 2)
Run III
(Example 3)
LOG10 VIABLE E.coli REMAINING (per ml)
TIME (days)
0
1
2
3
4
5
6
7
8
9

Fig. 2

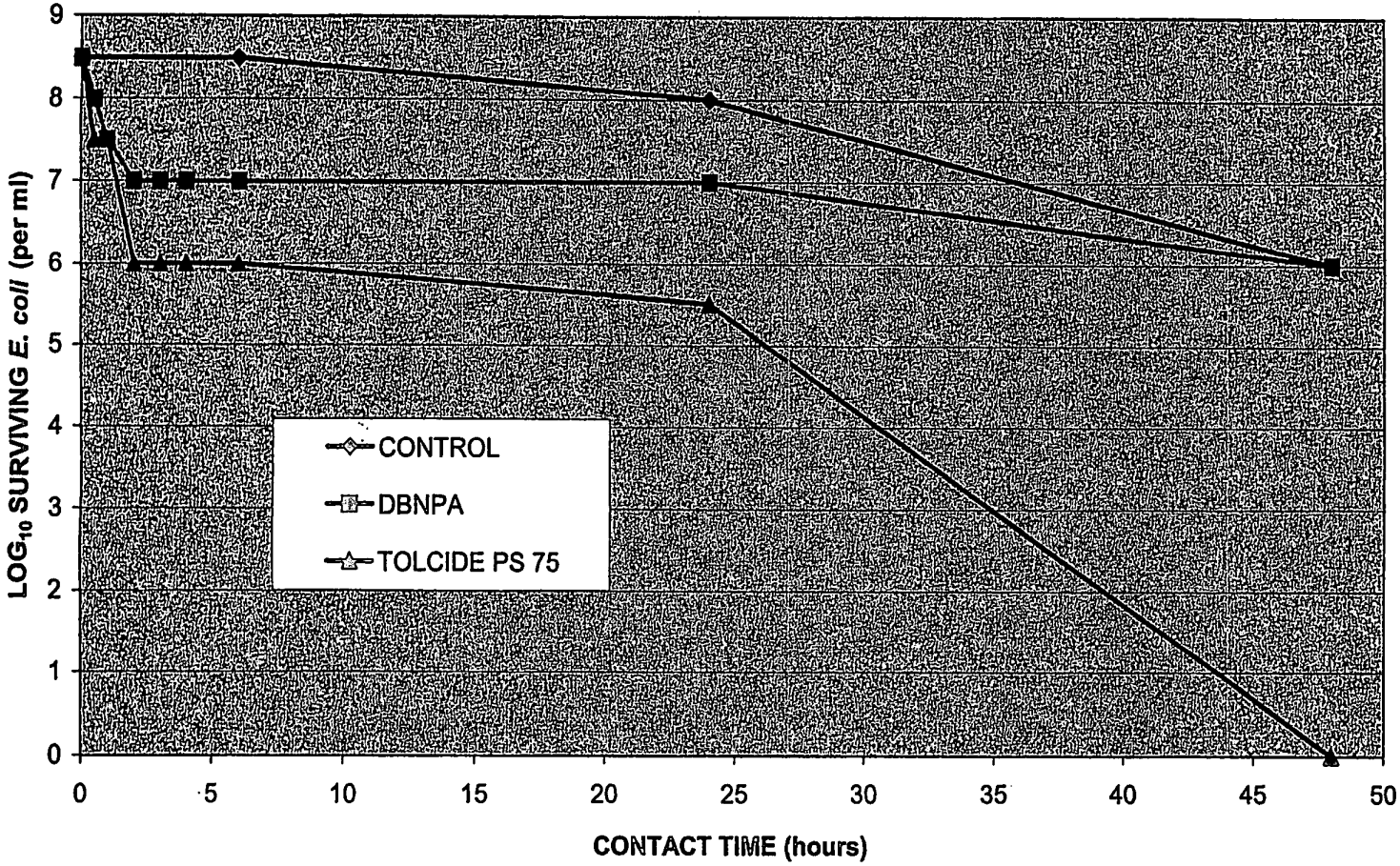
9
8
7
6
5
4
3
2
1
0
LOG10 SURVIVING E. coli (per ml)
CONTROL
DBNPA
TOLCIDE PS 75
0
5
10
15
20
25
30
35
40
45
50
CONTACT TIME (hours)

Fig. 3